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AN AUTOMATED MULTI-MODAL SERIAL SECTIONING SYSTEM FOR CHARACTERIZATION OF GRAIN-SCALE MICROSTRUCTURES IN ENGINEERING MATERIALS (PREPRINT)

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14. ABSTRACT

This paper describes the development of a new serial sectioning system that has been designed to collect microstructural, crystallographic, and chemical information from volumes in excess of 1 mm3. The system integrates a robotic multi-platen mechanical polishing system with a modern SEM that enables the acquisition of multi-modal data—scanning electron images, EBSD and hyperspectral EDS map—at each section. Selected details of the system construction as well as an initial demonstration of the system capabilities are presented.

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serial sectioning, 3D microscopy, EBSD, EDS

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An Automated Multi-Modal Serial Sectioning System for Characterization of Grain-Scale Microstructures in Engineering Materials

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Keywords: serial sectioning, 3D microscopy, EBSD, EDS

Abstract

This paper describes the development of a new serial sectioning system that has been designed to collect microstructural, crystallographic, and chemical information from volumes in excess of 1 mm^3. The system integrates a robotic multi-platen mechanical polishing system with a modern SEM that enables the acquisition of multi-modal data—scanning electron images, EBSD and hyperspectral EDS map—at each section. Selected details of the system construction as well as an initial demonstration of the system capabilities are presented.

Introduction

For the past two decades the materials research community has sustained research and development of hardware and software to enable 3D characterization of microstructural features across the wide range of length scales that are observed in many engineering materials. The list of dedicated laboratory-scale instrumentation for structural materials includes serial sectioning devices such as those based on micromilling [1], mechanical polishing [2,3], and femtosecond laser ablation [4], the 3D Atom Probe [5], x-ray microtomography systems, as well as instruments that have been adapted for this purpose like dual-beam Focused Ion Beam Scanning Electron Microscopes (FIB-SEM) [6] and Transmission Electron Microscopes outfitted with specialty tomographic sample holders.

Nonetheless, there remain capability gaps with regards to the type of data and the spatial coverage/resolution that can be achieved with the aforementioned list of current instrumentation. The 3D characterization system described in this paper attempts to fill one of these gaps through the collection of multi-modal information (local crystallographic, chemical, and image data) over a key range of length scales with regards to optimizing structural material performance, i.e., from micrometer-to-centimeter scale volumes.

The development of the system described herein was motivated by the success of FIB-SEM microscopes to rapidly characterize complex engineering alloy microstructures. These versatile microscopes have been used to perform serial sectioning experiments on a host of disparate material types: metals, ceramics, polymers, electronic materials, and biological materials. One of the key capabilities of FIB-SEM microscopes is the ability to incorporate advanced analytical

methods such as Electron BackScatter Diffraction (EBSD) and Energy Dispersive Spectrometers (EDS) systems. These methods provide more information regarding the local material state, and importantly, allow for segmentation of grains or second phases from complex microstructures using physically-meaningful parameters, such using a misorientation threshold to aggregate neighboring voxels into grains [7]. However, the limited material removal rate of liquid-metal ion sources has restricted the role of FIB-SEM microscopes to the study of nano- and micrometer-scale features in volumes that have dimensions on the order of tens-of-micrometers.

The multi-modal serial sectioning device described in this paper addresses the issue of volumetric coverage by replacing ion-beam sectioning with automated mechanical lapping and polishing. Mechanical polishing methods have been the traditional pathway for preparing the surface of materials for quantitative 2D microstructural analysis, and such methods have also been commonly employed for manual 'macro-scale' serial sectioning experiments [8], including those that incorporate multi-modal data [9]. Note that for the intended application there is the critical issue of preparing samples with minimal subsurface damage because of the stringent surface finish requirements of EBSD-based analysis. Unlike other automated serial sectioning instruments that employ mechanical polishing and optical microscopes [2,3], the device described in this paper has the ability to use multiple polishing platens to sequentially remove material using successively finer polishing media, thus enabling the use of EBSD. Although the device is still under development and full integration of the individual sub-systems is still in-progress, the basic layout of the device and a demonstration of its capabilities are presented in the following sections.

System Design and Capabilities

The initial system configuration consists of a three primary subsystems that reside within a 15' x 12' safety enclosure: a Mitsubishi transfer robot, a RoboMet.3D serial mechanical polishing instrument, and a Tescan SEM outfitted with EBSD and EDS detectors. An image taken from outside of the enclosure that shows the relative position of the three subsystems is shown in Figure 1.

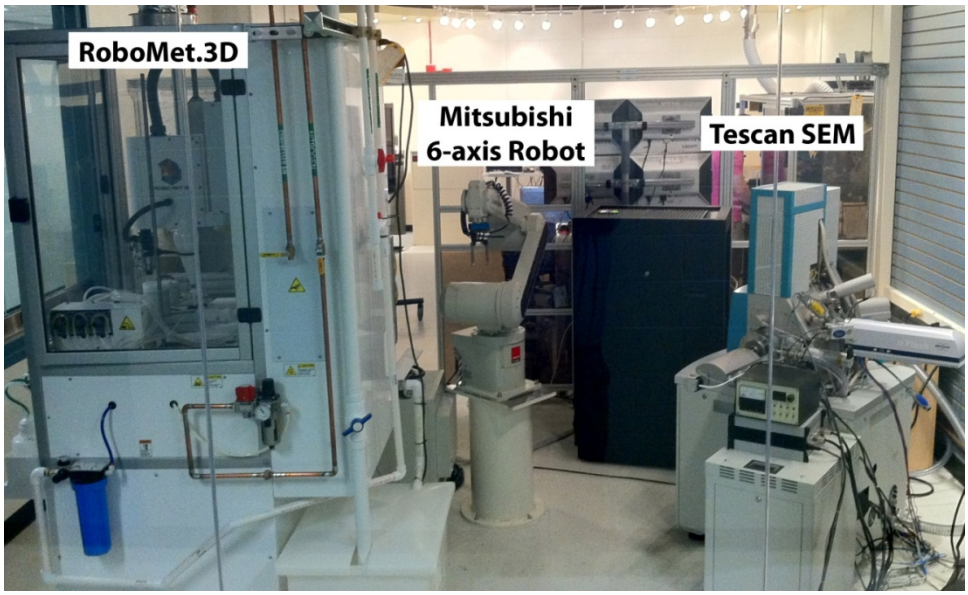

Figure 1. Image that shows the arrangement of the three main sub-systems that comprise the serial sectioning instrument.

These subsystems are controlled by a master computer that dictates the sequence of operations for the various sub-systems, which will also be eventually responsible for storing the multi-modal and all other metadata into a single 3D data file in HDF5 format [10]. The followings sections describe the key features for each of the three main sub-systems.

The Mitsubishi RV12SVL 6-axis robot arm has a 54'' reach, which allows it to readily move a 2" diameter stainless steel sample holder, Figure 2A, between sample exchange points on the Robo-Met.3D, the Tescan SEM, and an additional sample transfer stand that enables the robot to switch between two gripping configurations, as shown in Figures 2B and 2C. Custom end-of-arm-tooling enables two gripping modes. The top-gripping mode is used to place the holder in the SEM load lock with the sample surface facing up, while the side-gripping mode is used to insert the holder in the RoboMet.3D with the sample surface facing down. Half-cylindrical locating features on the top and bottom surfaces of the sample holders are used with mating features at the sample transfer positions ensure that the sample orientation and position is roughly maintained during hand-offs. In addition, the Mitsubishi robot will also be used to completely immerse the sample holder into beakers containing ethanol for ultrasonic cleaning, followed by articulating the sample in front of a nitrogen gas nozzle to dry the sample prior to SEM analysis. Robot arm movements are controlled remotely using executable scripts written in a manufacturer-supplied programming language (RTToolbox2). The flexibility for re-programming the motion of the 6-axis robot allows for easy reconfiguration of the characterization system in the future, such as adding additional sample preparation or analysis stations.

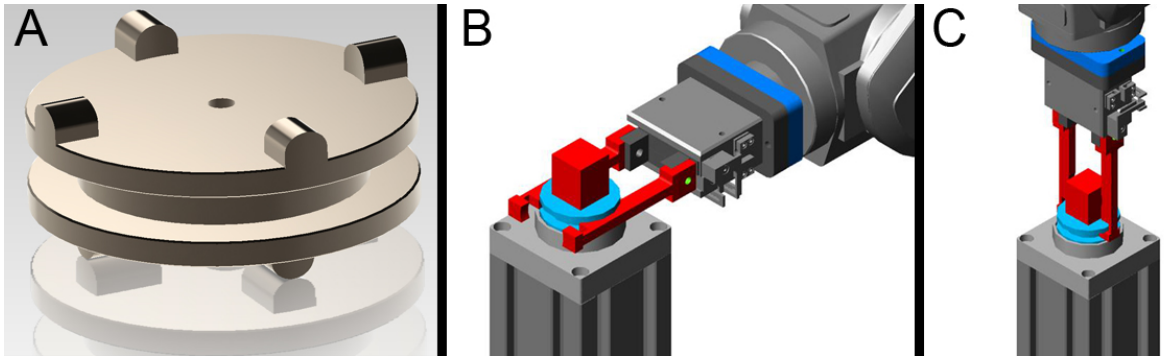

Figure 2. A) Schematic of the sample holder, B) Side-gripping mode for the Mitsubishi robot and end-of-arm-tooling, C) Top-gripping mode.

RoboMet.3D (www.ues.com/content/robomet3d), shown in Figure 3, is used to automatically perform the serial mechanical polishing step, removing a planar section of material while obtaining a high-quality surface finish that is suitable for both imaging and EBSD characterization in the SEM. The RoboMet.3D installed on this system has been customized with a multi-platen exchange arm, which allows the use of up to 8 platens in the polishing cycle. At present, at least two platens are used for EBSD-inclusive experiments, where typically a 1 micron diamond media along with a low-nap cloth is used to minimize differential polishing during the majority of material removal (for ~ 1 micrometer sections). This step is followed by sub-50 nm polishing media on a high-nap cloth, either colloidal silica or a two-step combination of alumina followed by colloidal silica, to minimize subsurface deformation. Water-soluble

polishing media and additional lubrication (distilled water) are automatically dispensed, and the application rates of all fluids as well as other polishing parameters such as wheel speed and sample oscillation are user-defined. Between polishing steps, current experiments use additional platens with high-nap cloths without any media to mechanically clean the surface of abrasives prior to ultrasonic immersion in an ethanol bath. Just before this cleaning step, the sample is dipped into a chemical well on the RoboMet.3D that is normally used for selective etching of the sample surface for optical microscopy. Rather than an etchant, the well is filled with diluted micro-organic soap to aid the cleaning process.

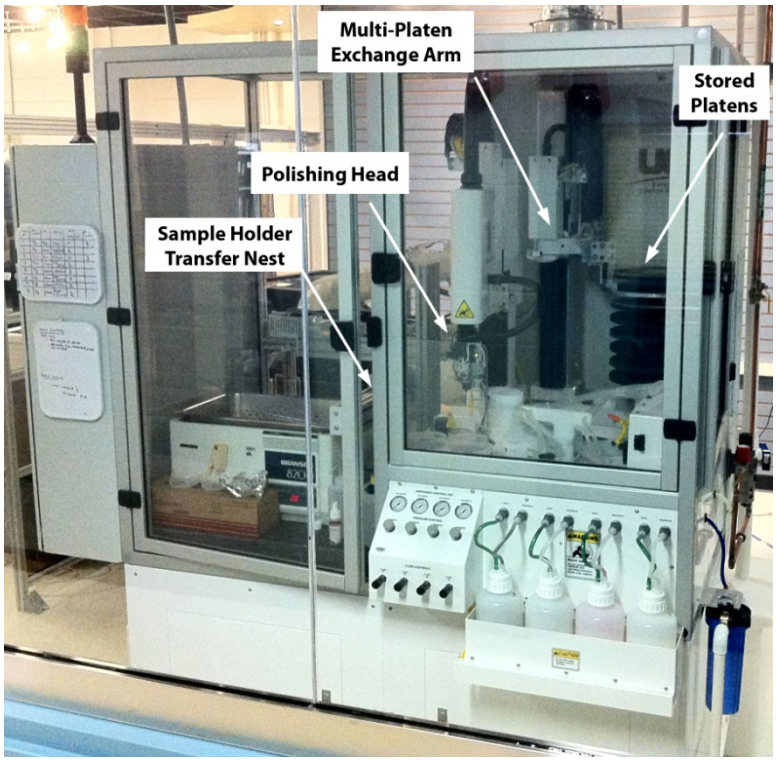

Figure 3. View of the front of the RoboMet.3D mechanical polishing system.

After mechanical polishing, a Tescan Vega 3 XMH microscope outfitted with a Bruker e-Flash 1000 EBSD and a X-Flash Quad 5040 EDS is used for characterization of the serial-section surface. An OEM-constructed customized robotic load lock assembly is used to transfer the sample holder into and out-of the SEM chamber. The cycle time for either insertion or removal takes approximately one minute, which significantly reduces the time needed to perform sample exchange. This process—as well as all other aspects of stage movement, detector insertion, beam optimization, image optimization, image collection, EDS and EBSD analysis—are controlled through the use of custom-developed Python scripts that use OEM-supplied application programming interfaces for the Tescan microscope (SharkSEM API) and the Bruker detectors (ESPRIT API).

To illustrate the type of algorithms that are used to control the operation of the microscope, the optimization of imaging conditions is performed using two scripts that determine the best focus and brightness/contrast values. At present, best focus is obtained by calculating the edge content within selected images over a user-defined range in focus settings:

$$\sum_{i=0}^{M}\sum_{j=0}^{N}\sqrt{S_x\left|I(i,j)\right|^2+S_Y\left|I(i,j)\right|^2} \tag{1}$$

$$S_x=\begin{bmatrix}-1 & 0 & 1\\ -2 & 0 & 2\\ -1 & 0 & 1\end{bmatrix}\quad S_y=\begin{bmatrix}-1 & -2 & -1\\ 0 & 0 & 0\\ 1 & 2 & 1\end{bmatrix} \tag{2}$$

where S_x and S_y are the Sobel kernels. A Gaussian curve is fit to the edge-content data as a function of focus/working distance, and the peak value from the fit corresponds to the best focus and the actual specimen-to-column working distance. The sample stage is raised or lowered to bring the sample surface to the desired working distance (e.g., 10 mm), and the focus is re-checked to confirm both accurate vertical positioning of the sample and optimal focus. The brightness and contrast of images are optimized using a very simple ad-hoc algorithm that compares the mean and standard deviation of the current image histogram to a user-defined reference image. The microscope brightness and contrast values are iteratively adjusted until the mean and standard deviation of the image closely match the reference values. At present, most of the algorithms used for automated instrument control similar to the latter example and are somewhat basic in design. Nonetheless, the images and data maps that are returned by the microscope are quite suitable for post-processing and analysis, although further advancement of these methods is desired. Figure 4 shows a representative backscattered electron image that has been collected without human intervention, which highlights the image quality that has been achieved to date.

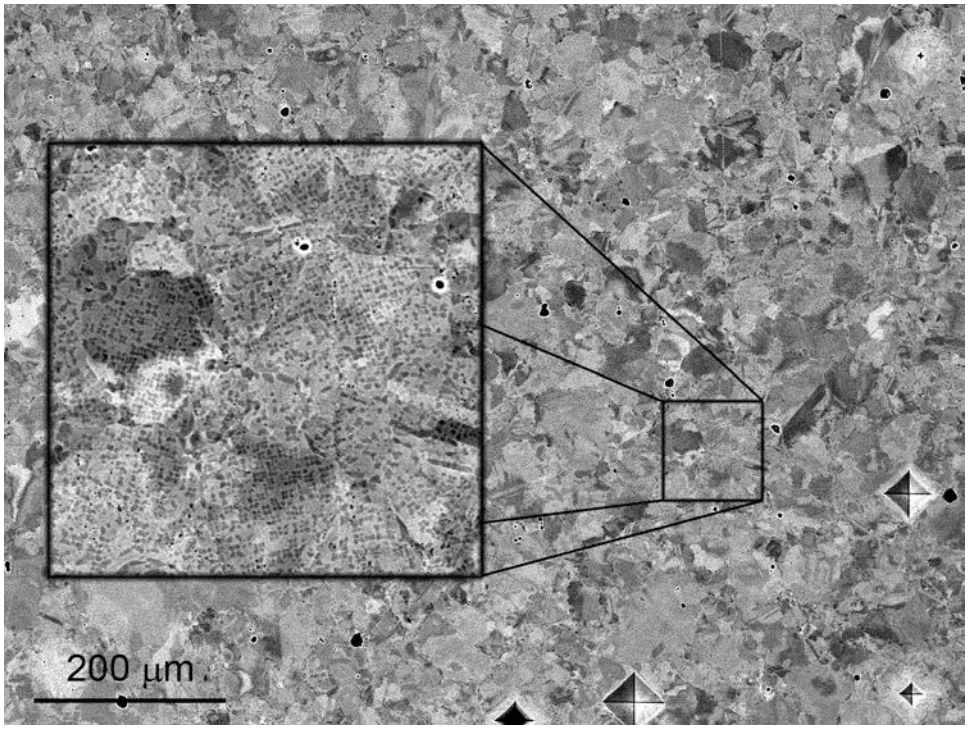

Figure 4. A representative backscattered electron image collected by the Tescan SEM using automation scripts. Field of view is 1 mm x 0.75 mm with pixel dimensions of 250 nm. Microscope operating conditions are an accelerating voltage of 30 kV, probe current of ~ 1 nA,

and a pixel dwell time of 100 μs. Material is an IN100 superalloy that has undergone a supersolvus heat treatment.

Table 1 provides a summary list of current capabilities for the multi-modal serial sectioning system. Desired improvements for the future include obtaining finer sectioning processes to directly overlap with meso-scale FIB-SEM experiments, and faster cycle times to improve data throughput. Reductions in the cycle time will come through both evolutionary improvements in instrument operation, for example, faster optimization of the electron imaging/analysis environment or minimizing the number of polishing pad operations, as well as by more radical methods, such as the use of real-time analysis to guide the efficient collection of EBSD and EDS data via sparse sampling. The volume of material that can be interrogated by the system can be easily in excess of 1 mm^3, and the main restriction on size is that the sample must clear the load-lock opening, which corresponds to a puck-shaped maximum volume of 38 mm diameter x 25 mm in length.

CAPABILITY	INITIAL	GOAL
SECTION THICKNESS	1 μm	200 nm
MIN. CYCLE TIME	> 20min	Faster
MATERIALS SYSTEMS	Ni, Ti Alloys	Most Alloys, Composites
IMAGING	SEI, BSE	Same
ORIENTATION INFO	Automated Collection	'Intelligent' Scans
CHEMICAL INFO	Automated Collection	'Intelligent' Scans

Table 1: Demonstrated and goal capabilities for the novel multi-modal serial sectioning system.

Example Application

As mentioned previously, the serial sectioning system is still in development and is not fully operational. However, each of the individual sub-systems can be operated in an automated fashion, and preliminary experiments have been conducted to help identify issues that need to be addressed during the final phase of development. These experiments have primarily consisted of performing semi-automated serial sectioning studies where the polishing and imaging sub-systems have been run without human intervention, while sample cleaning and microhardness indentation have been performed manually. Microhardness indents are used to 'frame' the area for microstructural analysis, and a few indents can be observed in the lower right portion of Figure 4. These features are useful as temporary reference marks that enable section-to-section registration that is unbiased by the morphology of internal microstructural features [11], as well as provide a measure of the local material removal rate [8].

Figure 5 shows a 3D reconstruction from a preliminary serial sectioning experiment of an IN100 superalloy that was previously described in Figure 4. The limited data set contains only 15 sections that have an average spacing of approximately 1 micrometer. Both BEI and EBSD maps have been collected at each section, and the total cycle time per section from polishing through SEM imaging and analysis was a little over two hours. Note that only the EBSD maps have been used to create the 3D reconstruction shown in Figure 5, which was generated using the DREAM.3D software environment (http://dream3d.bluequartz.net). This data set is too small to perform an unbiased analysis of the grain structure, as almost all of the grains touch one of the bounding surfaces of the data volume. However, once development of the automated serial sectioning system is completed, such analysis should become commonplace as suitable volumes could be collected within a few weeks, and the system will be capable of processing multiple samples at the same time.

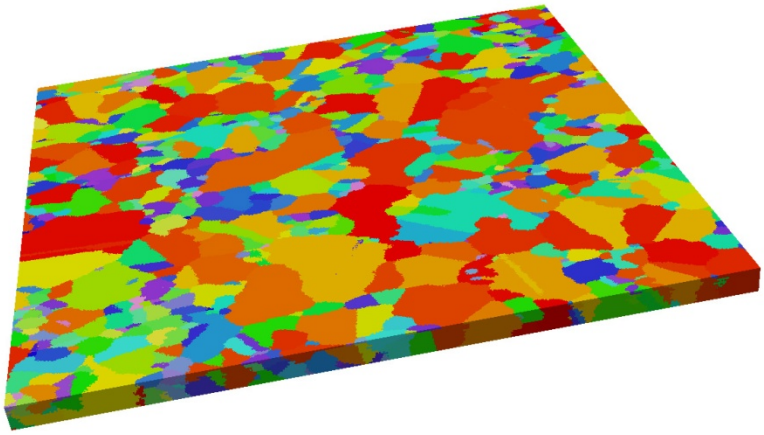

Figure 5. 3D reconstruction of an IN100 superalloy microstructure that was interrogated by the multi-modal 3D characterization system. Volume is 500 x 500 x 15 micrometers in size, and grain coloring corresponds to a unique grain ID.

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